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DERWENT-WEEK: 200230

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TITLE: Metal oxide coat formation use
coating liq. for semiconductor protection - liq.
comprises alkoxy silane,
tetra:alkoxy titanium cpd., and basic
zirconium cpd.,
opt. hydrolysed and mixed in organic
solvent

PATENT-ASSIGNEE: NISSAN CHEM IND LTD[NISC]

PRIORITY-DATA: 1991JP-0289848 (November 6, 1991)

PATENT-FAMILY:

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APPLICATION-DATA:

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ABSTRACTED-PUB-NO: JP 05124818A

BASIC-ABSTRACT:

A metal oxide coat formation use coating liq. comprises component (a) made of an alkoxy silane of formula $R_1Si(OR_2)_3-n$ (1) (R_1 is alkyl, alkenyl, or aryl, R_2 is alkyl; and n is 0 or 1), component (b) made of a tetra alkoxy titanium of formula $Ti(OR_3)_4$ (2) (R_3 is alkyl), component (c) made of a basic zirconium salt. The hydrolysed compound of the component (a), (b), and (c) or the hydrolysed compound of components (b), and (c) are mixed and dissolved in an organic solvent.

Here, the total of the component (a) in terms of SiO_2 , the component (b) in terms of TiO_2 , and the component (c) in terms of ZrO_2 , is pref 1-15 wt.%.

USE/ADVANTAGE - A dense metal oxide coating film having high hardness and good chemical resistance can be produced by heating and curing the coating liq. of the present invention. Used e.g. for making protective films for semiconductors.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: METAL OXIDE COAT FORMATION COATING LIQUID
SEMICONDUCTOR PROTECT
LIQUID COMPRISE ALKOXY SILANE TETRA ALKOXY
TITANIUM COMPOUND BASIC
ZIRCONIUM COMPOUND OPTION HYDROLYSIS MIX
ORGANIC SOLVENT

DERWENT-CLASS: G02 L02 L03 U11

CPI-CODES: G02-A05B; L04-C12A;

EPI-CODES: U11-C05B9;

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C1993-088916

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Notes:

1. Untranslatable words are replaced with asterisks (****).
2. Texts in the figures are not translated and shown as it is.

Translated: 07:33:11 JST 12/01/2007

Dictionary: Last updated 11/16/2007 / Priority: 1. Electronic engineering

TECHNICAL FIELD

[Industrial Application] This invention is applied to base materials, such as glass, metal, Ceramics Sub-Division, and plus CHIKKUSU, and relates to the coating liquid which can form the oxide tunic excellent in chemical resistance easily by carrying out heating hardening.

[Translation done.]

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Dictionary: Last updated 11/16/2007 / Priority: 1. Electronic engineering

CLAIMS

(57) [Claim(s)]

[Claim 1] (a) A component, a following general formula [1]

$R_1nSi(OR_2)_{4-n}$ [1] (R_1 expresses an alkyl group, an alkenyl group, and an aryl group, R_2 expresses an alkyl group, and n expresses the integer of 0 or 1.)

Alkoxysilane and (b) which are come out of and shown A component, following general formula [2]

$Ti(OR_3)_4$ [2]

(R_3 expresses an alkyl group.)

the tetra-alkoxy titanium come out of and shown, and (c) in a component and basic

JIRUKONIUMU salt -- (a) the hydrolysis thing of a component, the hydrolysis thing of the (b) component, and the (c) component -- or coating liquid for metal oxide tunic formation in which the hydrolysis thing of the (b) component and the (c) component carry out the mixed dissolution at an organic solvent.

[Claim 2] In Claim 1, it is a component (a). Composition of (b) and (c) [a molar ratio] (a) -- $/(a) + (b) + (c)$ -- the coating liquid for metal oxide tunic formation characterized by being $= 0 - 0.9$, $(b)/[(a) + (b) + (c)] = 0.05 - 0.95$ and $(c)/[(a) + (b) + (c)] = 0.05 - 0.2$.

[Claim 3] In Claim 1, it is (a). They are SiO_2 and (b) about the alkoxysilane of a component. They are TiO_2 and (c) about tetra-alkoxy titanium of a component. Coating liquid for metal oxide tunic formation which converts the basic JIRUKONIUMU salt of a component into ZrO_2 , respectively, and is characterized by the total being 1 to 15 weight %.

[Claim 4] In Claim 1, it is (c). Coating liquid for metal oxide tunic formation characterized by the basic JIRUKONIUMU salt of a component being a basic zirconium nitrate.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention is applied to base materials, such as glass, metal, Ceramics Sub-Division, and plus CHIKKUSU, and relates to the coating liquid which can form the oxide tunic excellent in chemical resistance easily by carrying out heating hardening.

[0002]

[Description of the Prior Art] [the coating liquid for oxide tunic formation / point / that an oxide tunic can be made to form in a large area easily] The alkali ion elution preventing film from the glass for liquid-crystal-display elements, the insulation protection film of a transparent conducting film, an orientation control film, the passivation film of IC and also boron, and a phosphorus are added, and it is widely used as hard-coat of a diffusion film, glass, the meal-proof protective film of a surface of metal, and a plastic. The coating liquid for SiO₂ tunic formation is mainly well known triggered by the stability of liquid, and the ease of manufacture.

[0003]

[Problem(s) to be Solved by the Invention] However, the SiO₂ above-mentioned tunic has a problem in alkali resistance, in order to form silicic acid alkali salt and to dissolve easily by existence of alkali ion, and it has further the fault of dissolving easily also to fluoric acid. It was not able to be used when a base material was processed by the case where alkali cleaning is carried out, or fluoric acid for the reason.

[0004] In order to raise the chemical resistance of SiO₂ tunic Adding the oxide of ZrO₂ and TiO₂ grade is known well, and [pottery-industry association magazine 85 volume 448 pages (1977)] Although there is illustration of the coating liquid which can form tetra-ethoxy silane, tetraisopropoxy titanium, and the tunic excellent in the alkali resistance which consists of the mixture of tetra-isopropoxy JIRUKONIUMU Since moisture in the air had hydrolyzed coating liquid given [this] in a pottery-industry association magazine after applying it on a base material, without hydrolyzing a metal ARUKOKISHIDO compound, there was a problem also in the stability of the upper coating liquid which has a problem in membrane formation nature.

[0005] On the other hand, a JP,63-145370,A number has illustration of the coating liquid which consists of tetra-alkoxysilane and basic JIRUKONIUMU salt, and basic salt-ized JIRUKONIUMU and a basic zirconium nitrate are mentioned as basic JIRUKONIUMU salt. receiving SiO₂ in ZrO₂, in order to raise the alkali resistance of SiO₂ tunic generally -- more than 20 mol % -- it is necessary to add In the above-mentioned JP,63-145370,A number, since basic salt-ized JIRUKONIUMU had the high solubility over organic solvents, such as alcohol, it was possible to have added ZrO₂ component of the quantity which fully discovers alkali resistance, but since a lot of chlorine was contained in coating liquid, there was a problem in respect of corrosiveness or purity. Moreover, the basic zirconium nitrate had the problem that ZrO₂ of quantity which needs the solubility over an organic solvent for SiO₂ tunic to fully have alkali resistance since it is low compared with basic salt-ized JIRUKONIUMU could not be

[Function] (a) of coating liquid (b) And (c) Component, The oxide tunic which shows the chemical resistance which was excellent in $(a)/[(a)+(b)+(c)] = 0 - 0.9$, $(b)/[(a)+(b)+(c)] = 0.05 - 0.95$ and $(c)/[(a)+(b)+(c)] = 0.05 - 0.2$ by a molar ratio is given. Although it is known that the compound tunic of TiO_2 , or SiO_2 and TiO_2 generally shows the outstanding alkali resistance and acid resistance compared with SiO_2 tunic, it is hard to say that the chemical resistance is also enough. Although it was known well that alkali resistance will improve sharply by on the other hand making SiO_2 component contain ZrO_2 component 20weight % or more, the problem was in acid resistance.

[0018] According to composition of above-mentioned this invention, Ti atom is inserted in SiO_2 frame, acid resistance improves, and when Zr exists in a frame by an ionic state further, large alkali-proof improvement is made. Since TiO_2 component shows chemical resistance high originally above and Zr component exists by an ionic state when the (a) component which is SiO_2 component is not included, still higher alkali resistance is shown.

[0019] (b) And (c) As for neither of component, the case of less than 0.05 shows sufficient chemical resistance by the above-mentioned molar ratio. Moreover, (c) Even if more components than 0.2 are contained by a molar ratio, chemical resistance does not improve any more, but it is (c) of this invention further. It is difficult for a component for a basic zirconium nitrate to be desirable and to make it dissolve mostly from 0.2 from the point which does not contain halogen. To all the ARUKOKISHIDO machines of an ARUKOKISHIDO compound, if there is less water used in the case of hydrolysis at a molar ratio than 0.2 time, hydrolyzing becomes inadequate, the monomer of an ARUKOKISHIDO compound remains so much, the membrane formation nature of coating liquid will worsen and its mechanical intensity of the tunic obtained will not improve, either. If more on the contrary than 2.5 times, the storage stability of coating liquid will become scarce and will cause the increase in viscosity of coating liquid, gelling, etc.

[0020] The coating liquid of this invention shows the stable membrane formation nature, without being influenced by moisture in the atmosphere, since water has already hydrolyzed. Moreover, there is no change of the coating liquid by moisture absorption, and it excels also in the stability of coating liquid in use. (a) of the coating liquid of this invention They are SiO_2 and (b) about a component. They are TiO_2 and (c) about a component. If a component is converted into ZrO_2 and there is $\text{SiO}_2 + \text{TiO}_2 + \text{ZrO}$ less 2 minute than 1 weight %, The thickness of the coat obtained by one application is thin, and many applications are not needed and efficient in order to obtain predetermined thickness.

[0021] On the other hand, if 15 weight % is exceeded, it will become thick, it will become difficult to obtain a uniform tunic, the storage stability of coating liquid will also become scarce, and the thickness of the coat obtained by one application will cause the increase in viscosity of coating liquid, gelling, etc.

[0022]

[Example] Ethanol 72.61g was put into the reaction flask equipped with work-example-1 condenser tube and the dropping funnel as the tetra-ethoxy silane 20.8g, tetraisopropoxy titanium 3.55g, and a solvent, and it mixed well. The solution which dissolved 3.34g of basic zirconium nitrate 2 hydration things in 5.4g of water and Ethanol 36.3g was dropped at this mixture under room temperature. According to dropping, about 8 degrees C of reaction liquid generated heat from 25 degrees C to 33 degrees C. Churning was continued for 1 hour and used as coating liquid. At 550 degrees C of this coating liquid, ignition residue is 6 weight % and Si:Ti:Zr was set to 8:1:1.

[0023] Coating liquid was manufactured by the same method as a work example 1 using the tetra-ethoxy silane (the inside TEOS of front, and notation) of the composition shown in work-example-2, 3, and 4 table-1, tetraisopropoxy titanium (the inside TIOT of front, and notation), and a basic zirconium nitrate 2 hydration thing (the inside ZrN of front, and notation). 550-degree C ignition residue was 6 weight %, respectively.

[0024] Ethanol 79.71g was well mixed with the tetramethoxy silane 7.6g, methyl trimetoxysilane 6.8g, and tetra-ethoxy titanium 2.85g as a solvent in the reaction flask equipped with work-example-5 condenser tube and the dropping funnel. The solution which dissolved 3.34g of basic zirconium nitrate 2 hydration things in 5.4g of water and Ethanol 36.3g was dropped at this mixture under room temperature. According to dropping, about 13 degrees C of reaction liquid generated heat from 25 degrees C to 38 degrees C. Churning was continued for 1 hour and used as coating liquid. At 550 degrees C of this coating liquid, ignition residue is 6 weight % and Si:Ti:Zr was set to 8:1:1.

[0025] Ethanol 154.9g was put into the reaction flask equipped with comparative example-1 condenser tube and the dropping funnel as the tetra-ethoxy silane 20.8g, tetraisopropoxy titanium 42.6g, and a solvent, and it mixed well. 0.2g of nitric acid and the solution which dissolved 13.5g of water in Ethanol 74.3g were dropped at this mixture under room temperature as an acid catalyst. According to dropping, about 15 degrees C of reaction liquid generated heat from 25 degrees C to 40 degrees C. Churning was continued for 1 hour and used as coating liquid. Ignition residue was 6 weight % at 550 degrees C of this coating liquid, and Si:Ti was 4:6.

[0026] Ethanol 72.96g was put into the reaction flask equipped with comparative example-2 condenser tube and the dropping funnel as the tetra-ethoxy silane 20.8g and a solvent, and it mixed well. The solution which dissolved 5.34g of basic zirconium nitrate 2 hydration things in 5.4g of water and Ethanol 36.5g was dropped at this mixture under room temperature. According to dropping, about 5 degrees C of reaction liquid generated heat from 25 degrees C to 30 degrees C. Churning was continued for 1 hour and used as coating liquid. At 550 degrees C of this coating liquid, ignition residue is 6 weight % and Si:Zr was set to 8:2.

表-2 被膜試験例

	膜厚 (10^{-8} cm)	耐アルカリ性 (10^{-8} cm/min)	耐酸性 (10^{-8} cm/sec)
実施例 1	1200	0	30
実施例 2	1300	0	20
実施例 3	1500	5	25
実施例 4	1200	0	30
実施例 5	1400	2	25
比較例 1	1300	20	50
比較例 2	1100	3	125
比較例 3	1000	80	250

[0034]

[Effect of the Invention] According to the coating liquid of this invention, the tunic which was excellent in alkali resistance and acid resistance compared with SiO₂ conventional tunic can be formed easily, and impurity ion, such as alkali and halogen, is not included in coating liquid. Therefore, it is suitable as protective films and insulating films, such as a liquid-crystal-display element which dislikes mixing of impurities including mostly the etching step by alkali cleaning, acid washing, or fluoric acid, and a semiconductor device.

[0035] Moreover, it is beforehand hydrolyzed by water, coating liquid is stable to moisture in the atmosphere, and the application of the dipping method and a flexo printing method with much time exposed to air atmosphere is possible.

[Translation done.]